5th NOVALIS meeting

Bundesministerium für Bildung und Forschung

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Tasks at Uni Siegen

1. Deposition studies:

   - **NbTiN** in CC800: DCMS and HiPIMS

   - Nb_3Sn in BoxCoater: RFMS, "test" the material, tests for SIS structures

   - **MgB_2** in PVD/SEY chamber: co-sputtering with RFMS

   on metal (Cu, Nb…) as well as insulating (AlN, TiO_2, Al_2O_3) substrates

2. Substrate preparation: **mechanical** and **electropolishing** of **Cu**

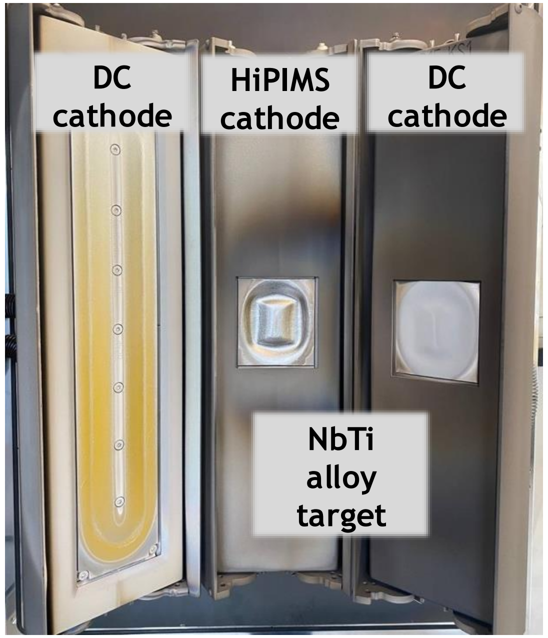
3. Sample characterization: film morphology, microstructure +

   PALS experiments (HZDR , Sebastian Klug, Oskar Liedke) +

   SC and RF properties (INFN-LNL Italy, Dorothea, Davide, Giovanni; IEE Slovakia, Eugen)

Experimental setup

NbTiN in commercial coating system CC800/9



- Target size: 100 x 88 mm^2
- Target: NbTi alloy **80:20 wt%** (99,95%, Robeko) ~$Nb_{0.67}Ti_{0.33}$ target composition
- Bake-out time: 6 h at 290°C
- Base pressure: ~6.0 x 10^{-7} mbar
- MF plasma etching for substrate plasma cleaning

Substrates

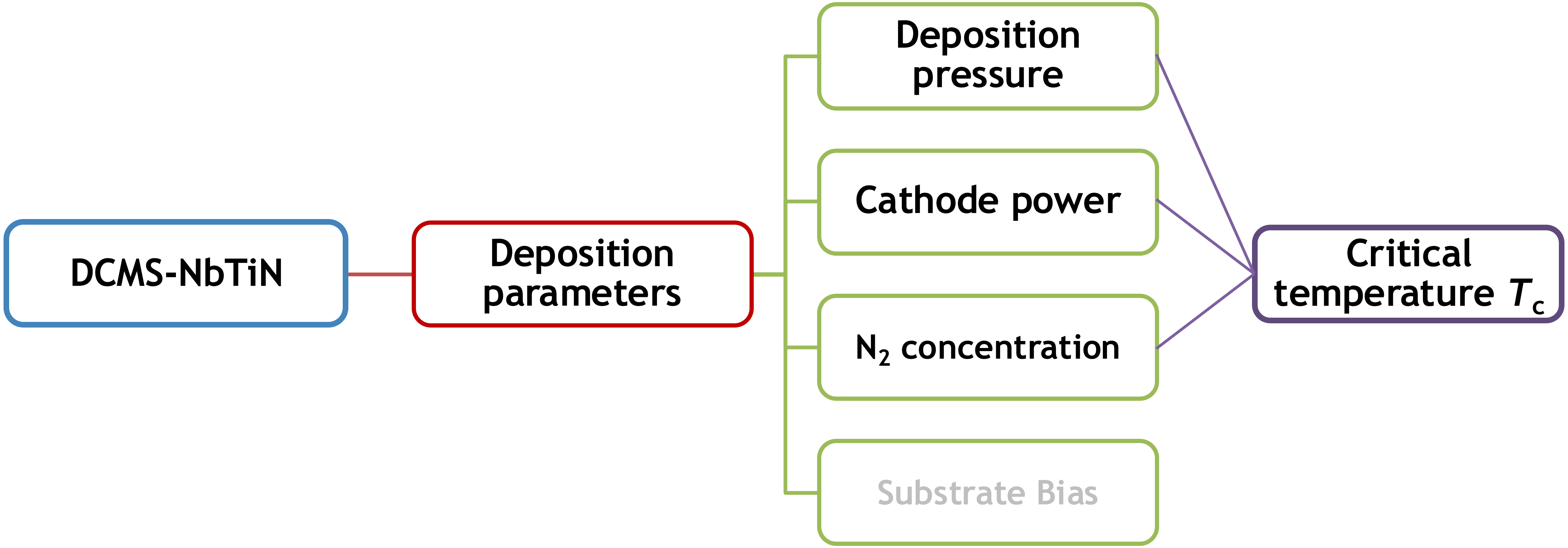
- p-doped Si<100>, 15 x 15 mm^2
- Polycrystalline OFHC Cu samples, 1 mm thick, 25 x 25 mm^2
- Sample treatment of Cu substrates: mechanical polishing + electropolishing in a solution of o-H_3PO_4 (85 %) and n-butanol (C_4H_9OH), 3:2 ratio

Characterization methods:

- SEM, EDX, XRD, AFM
- T_c (coil-induction) measurement station[1] on Si samples

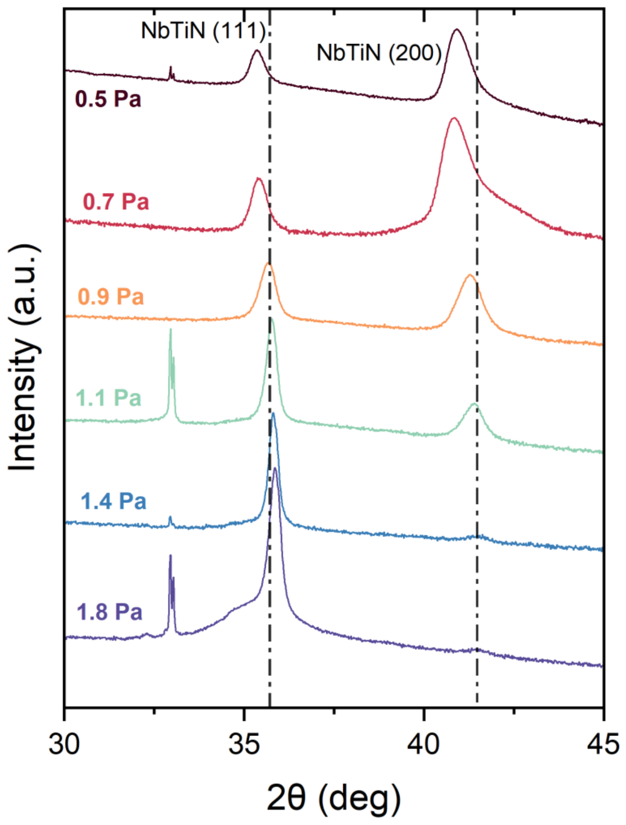
[1] D. Fonnesu et al., *in Proc. SRF'21*, East Lansing, MI, USA, Jun.-Jul. 2021, pp. 105-108 doi.org/10.18429/JACoW-SRF2021-SUPFDV018

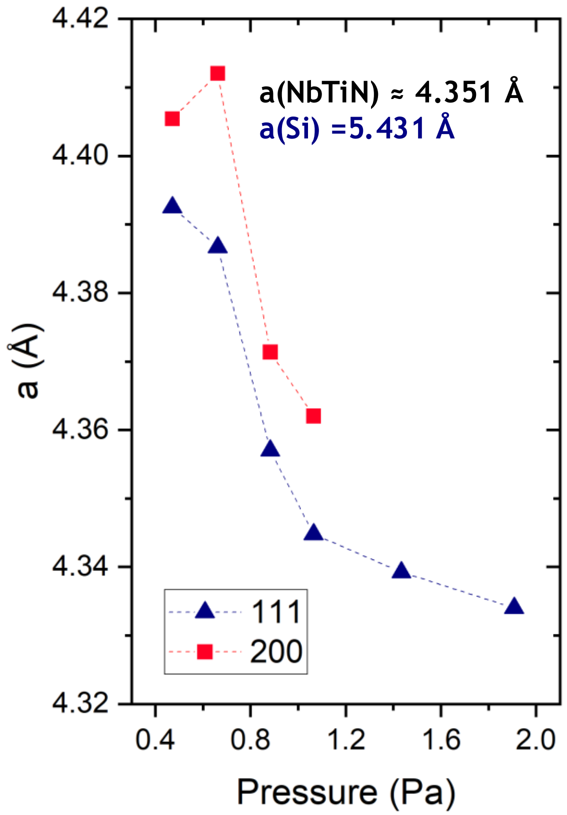
Experimental plan

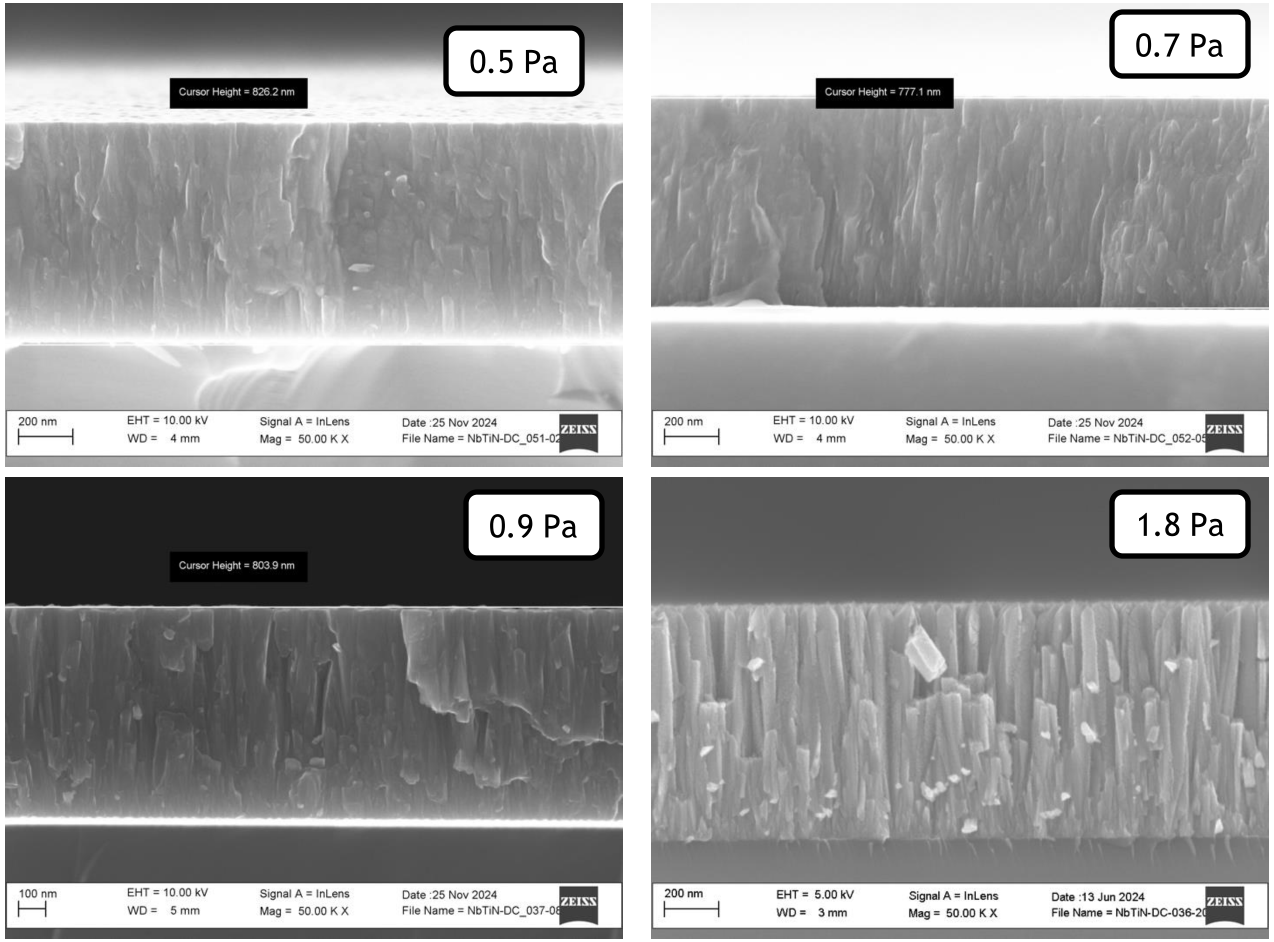


DCMS-NbTiN

Effect of deposition pressure



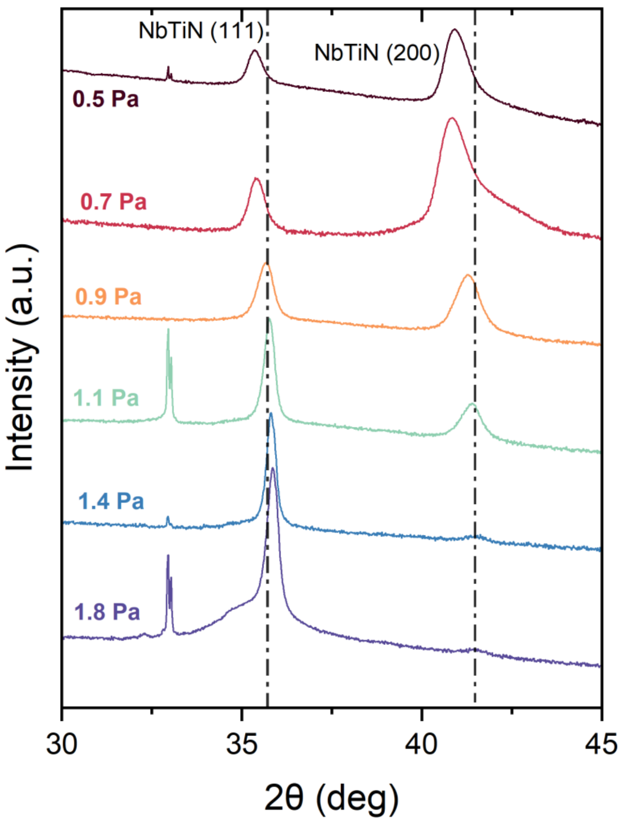


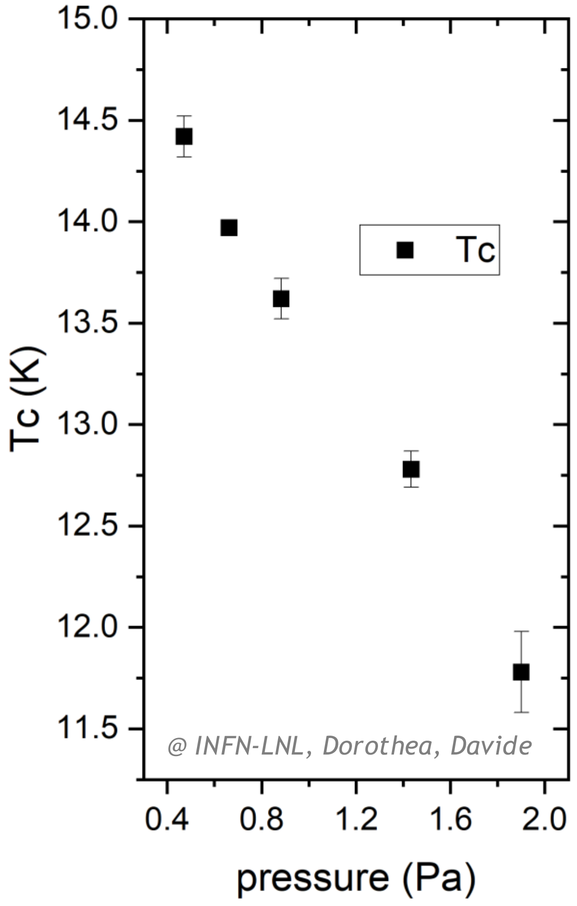


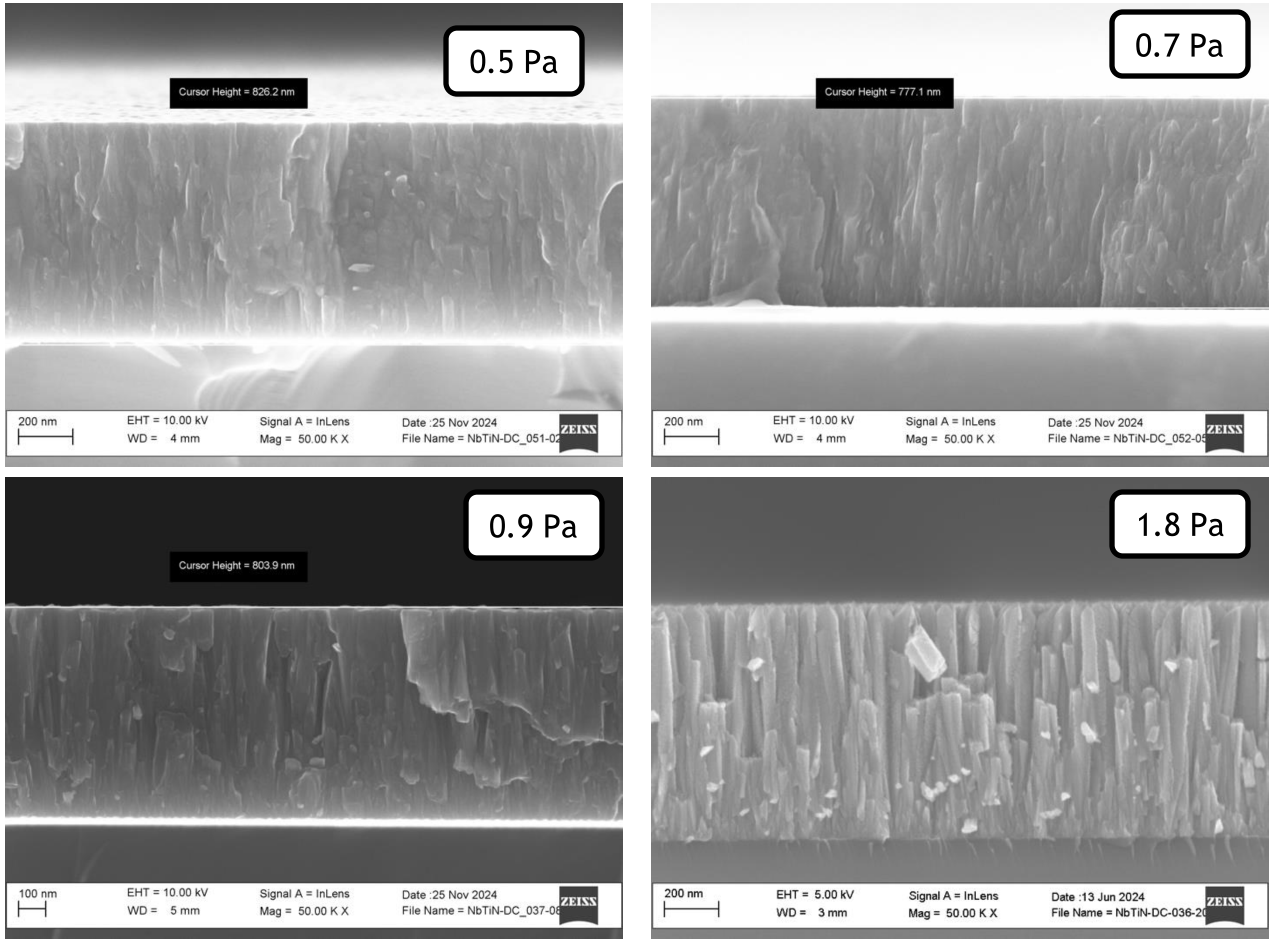
Deposition conditions:
P_{target} = 400 W, N_2 = 9%,Bias = -50V

DCMS-NbTiN

Effect of deposition pressure





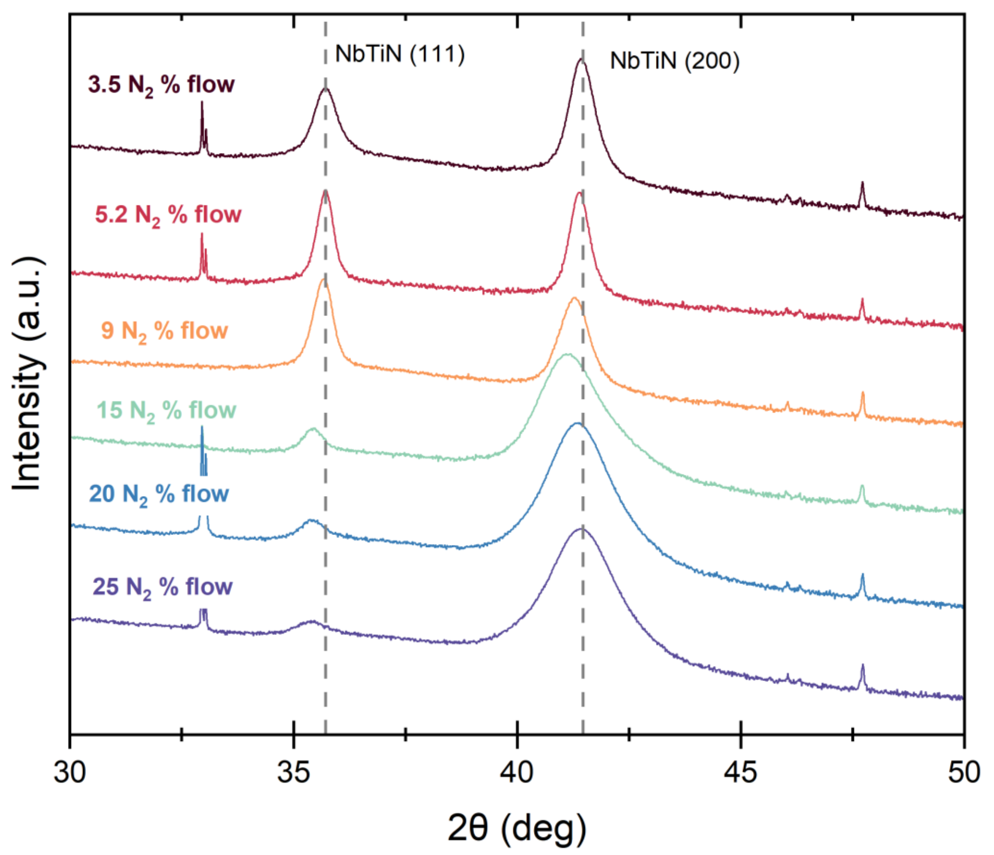


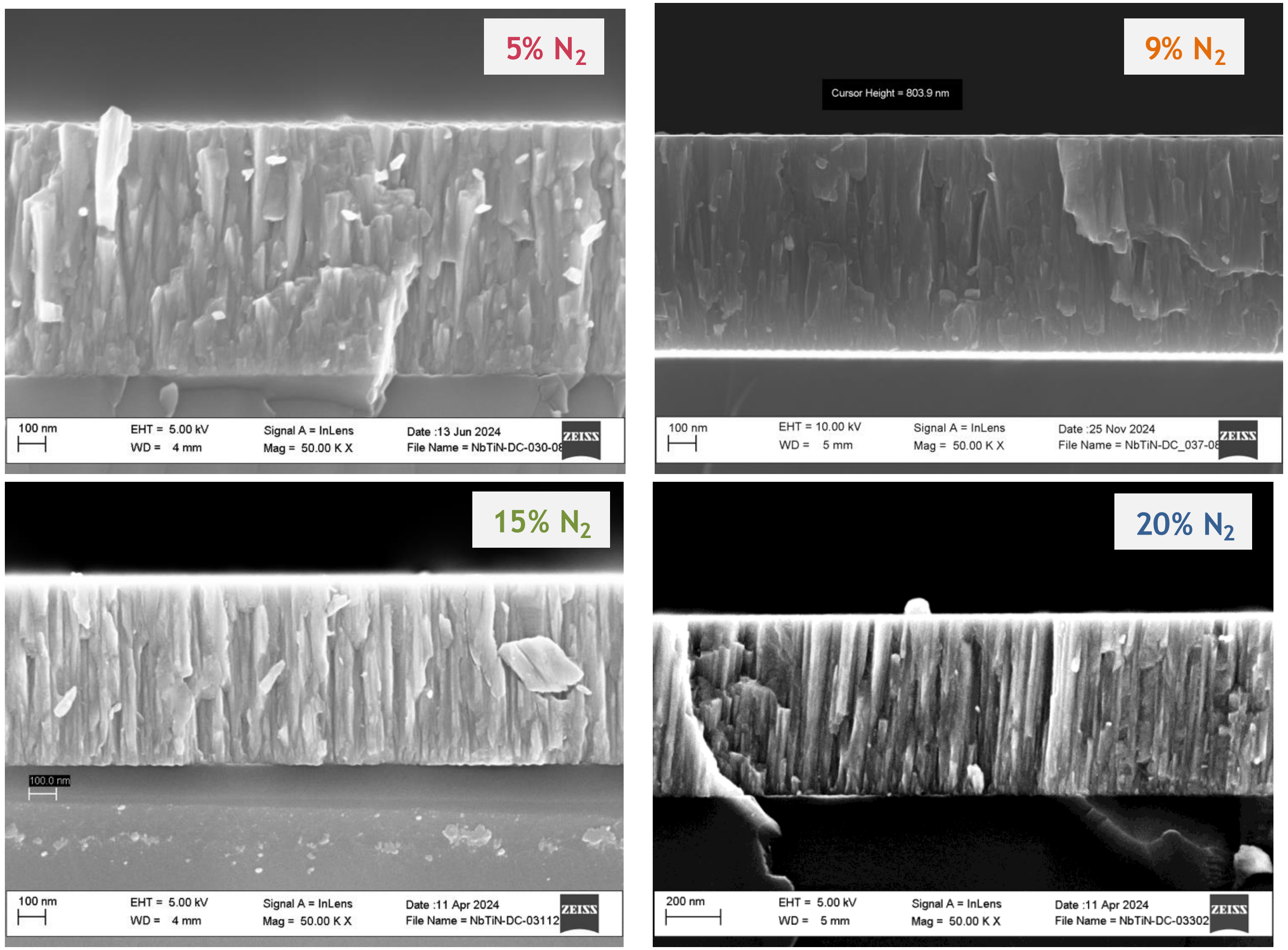
Deposition conditions:
P_{target} = 400 W, N_2 = 9%,Bias = -50V

DCMS-NbTiN

Effect of N_2 concentration

Deposition conditions: P = 400 W, p_{dep} = 0.9 Pa, Bias = -50V

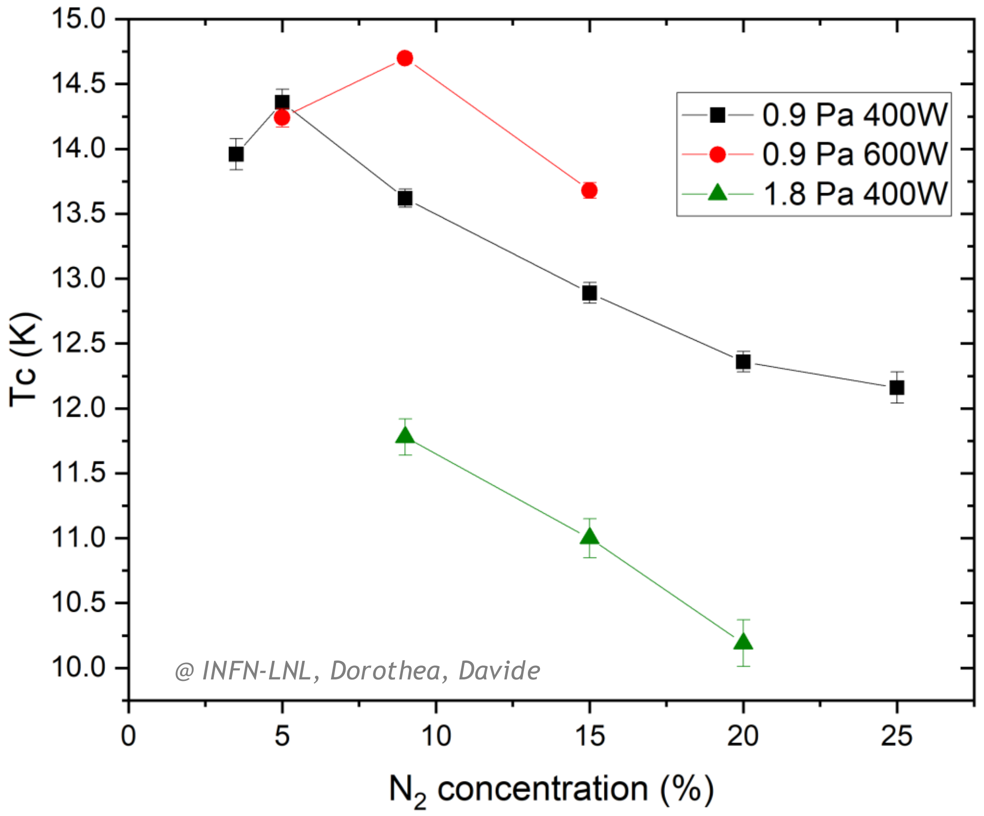


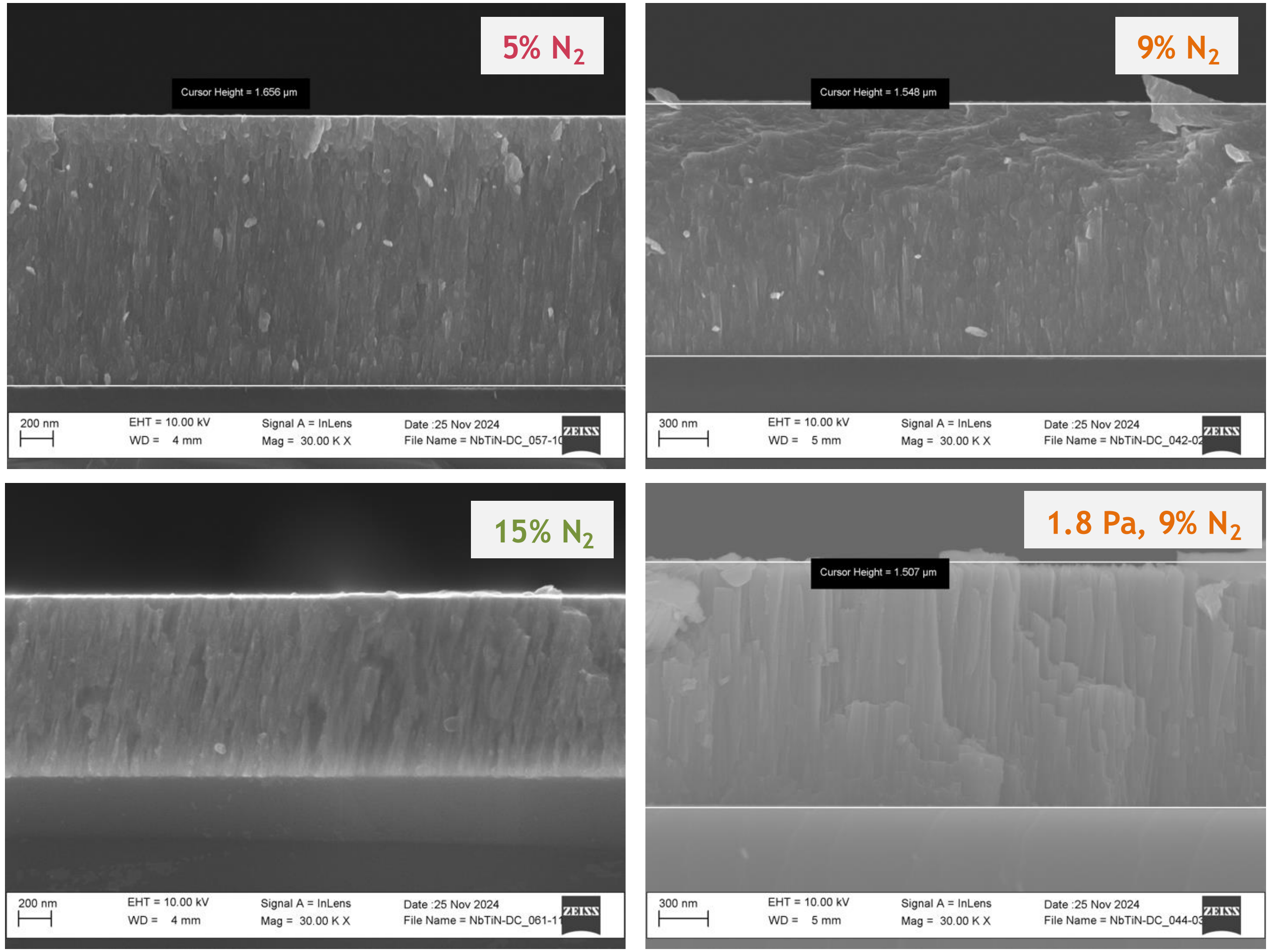


DCMS-NbTiN

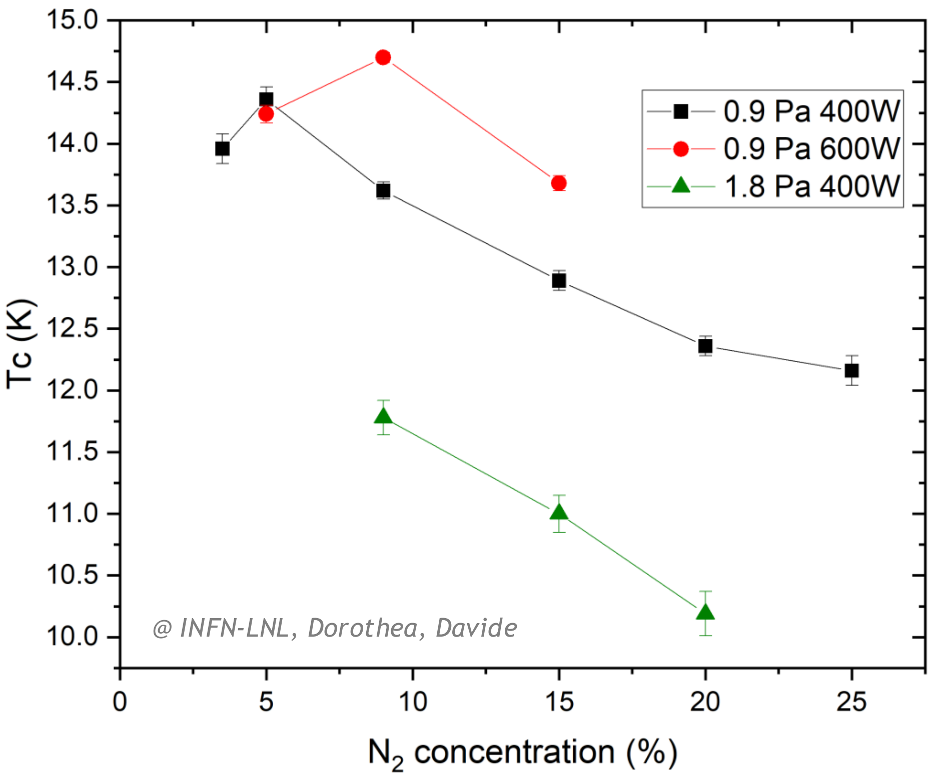
Effect of N_2 concentration and cathode power

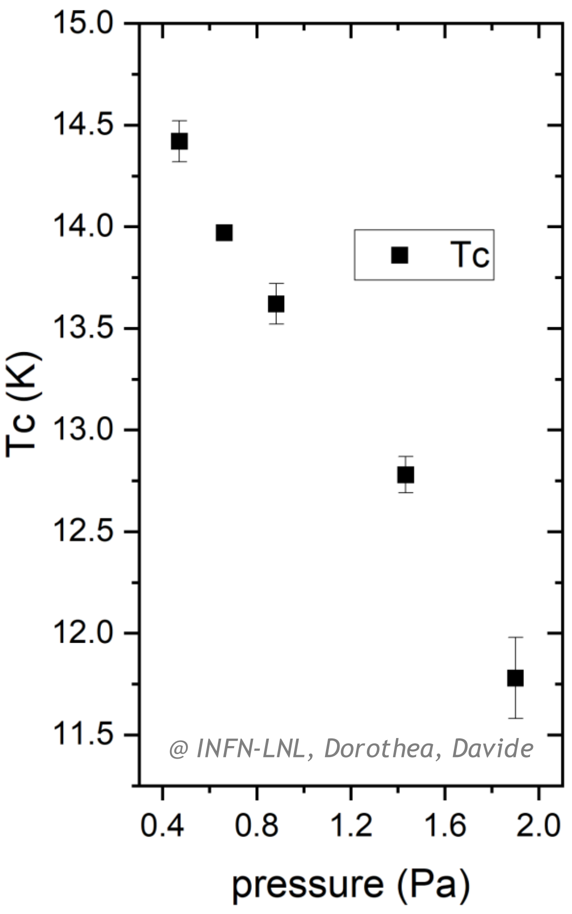
Deposition conditions: **P = 600 W**, p_{dep} = 0.9 Pa, Bias = -50V





DCMS-NbTiN



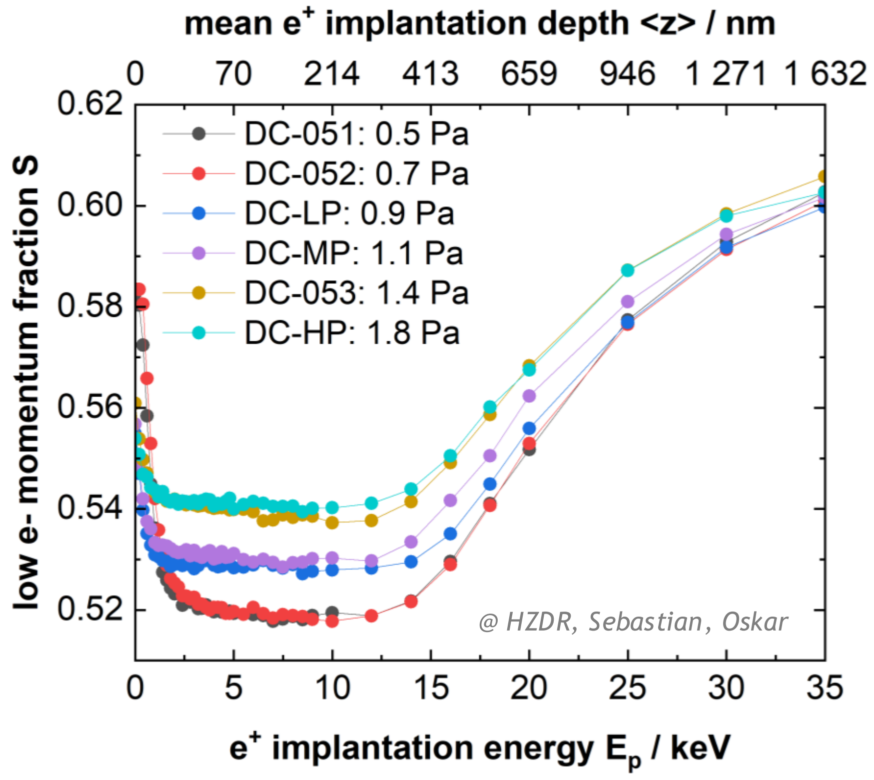


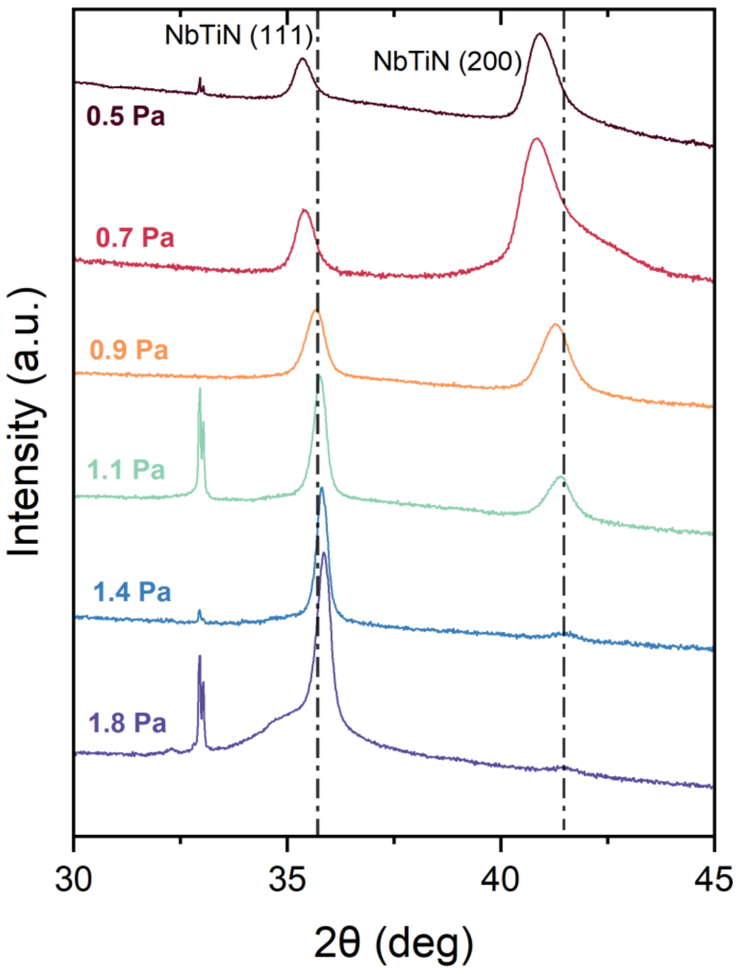
High T_c - where?

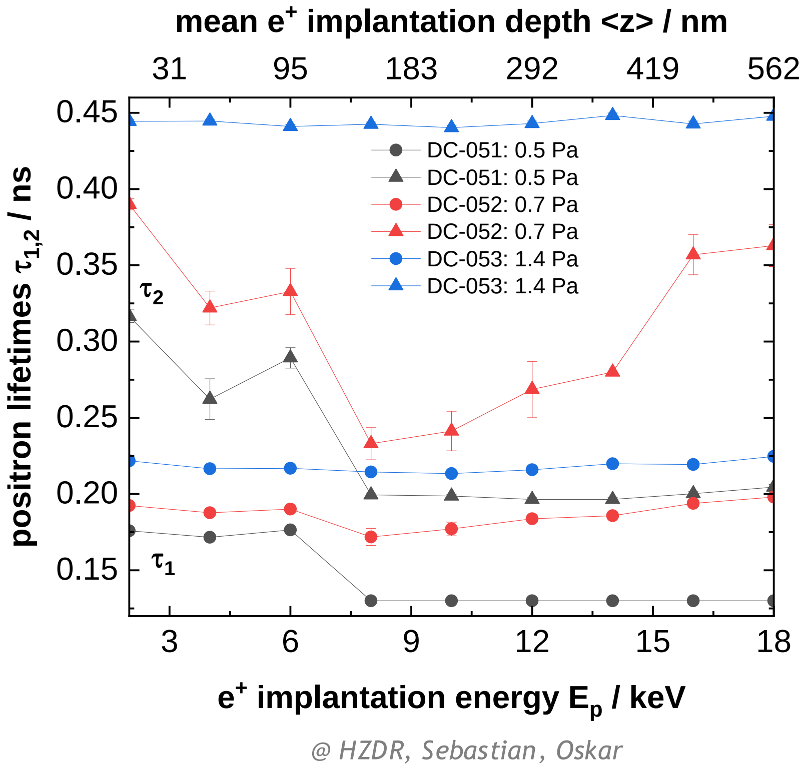
- **Low deposition pressure:** 0.5-0.7 Pa
- **High cathode power:** 600W (6.8 W/cm^2)
- **Low N_2 flow:** <9%, related to the cathode power
- **Deposition temperature:** 250°C → probably too low

DCMS-NbTiN

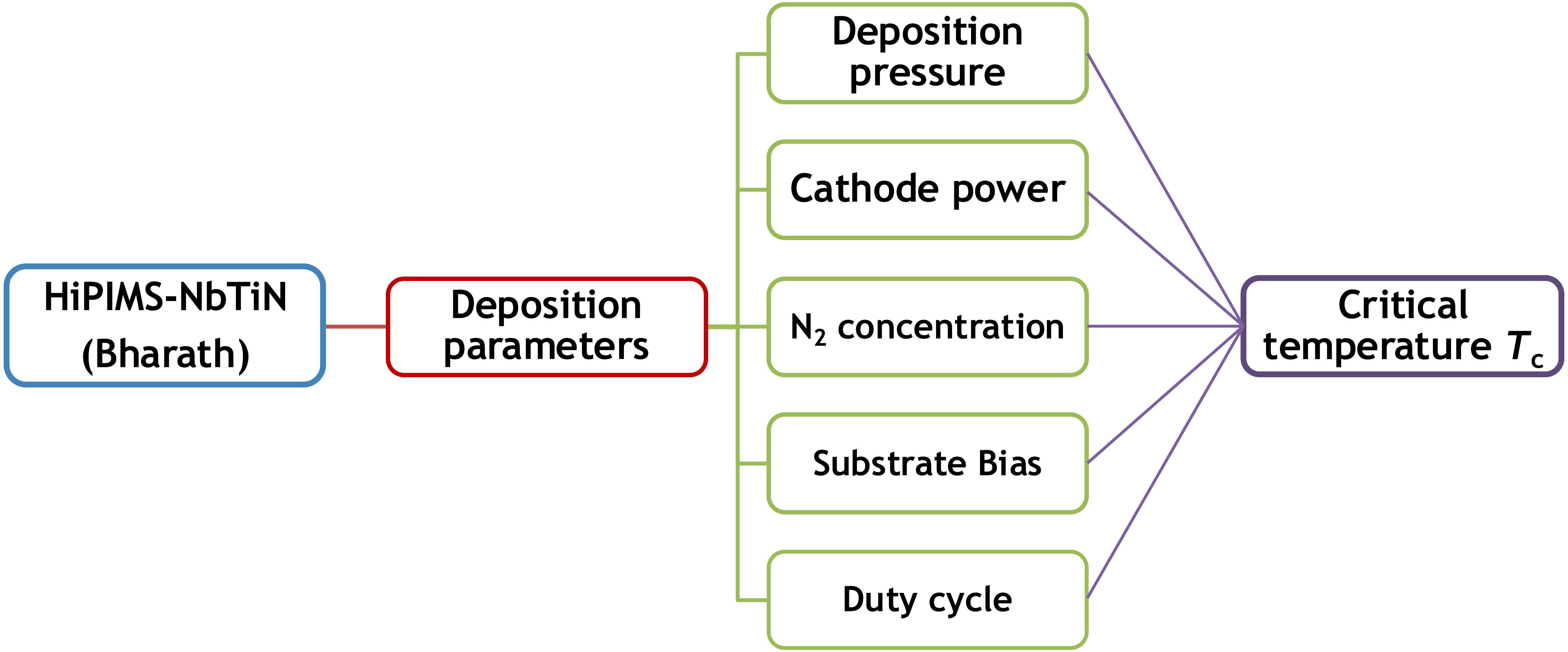
PALS experiments: deposition pressure





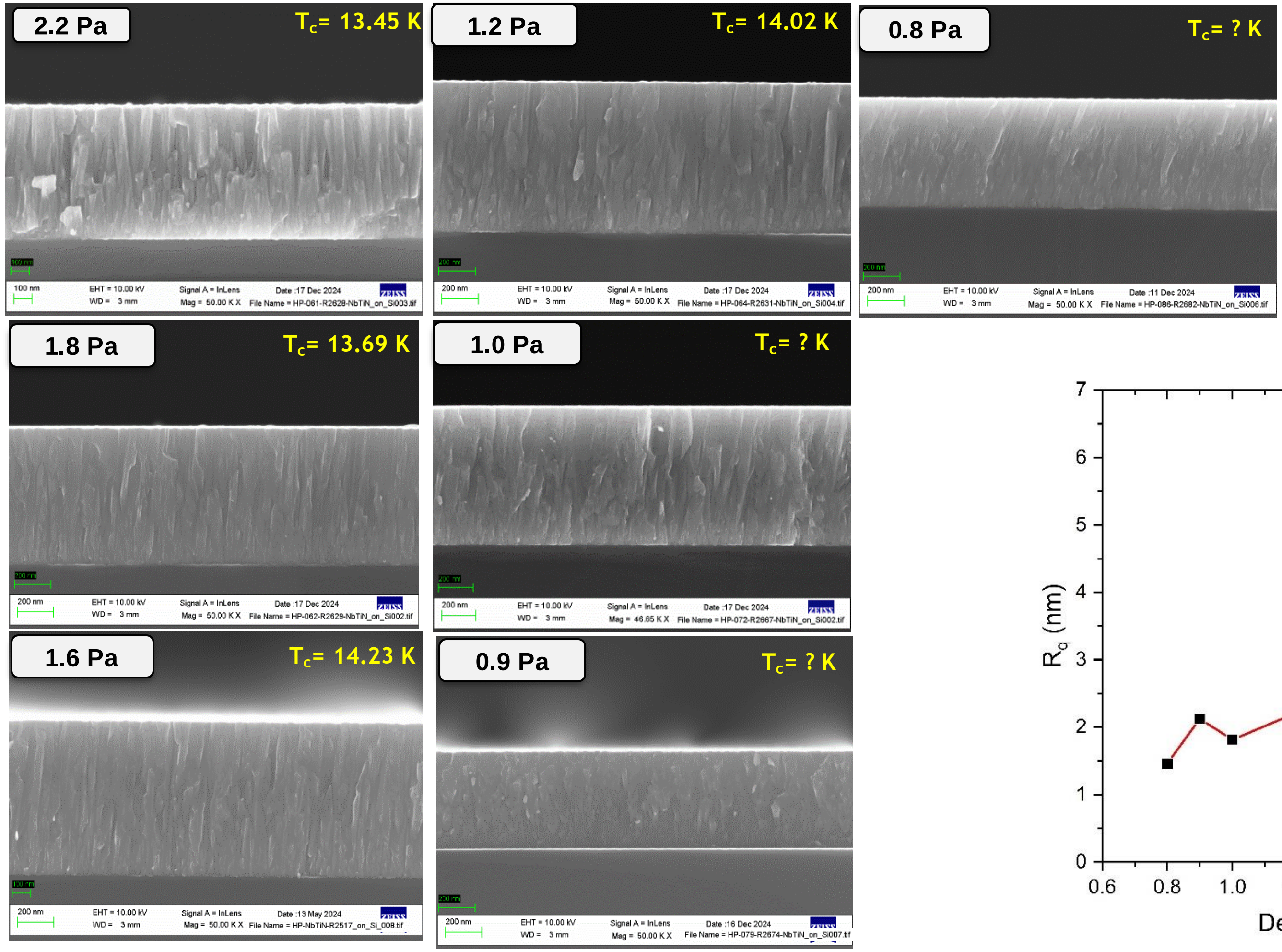


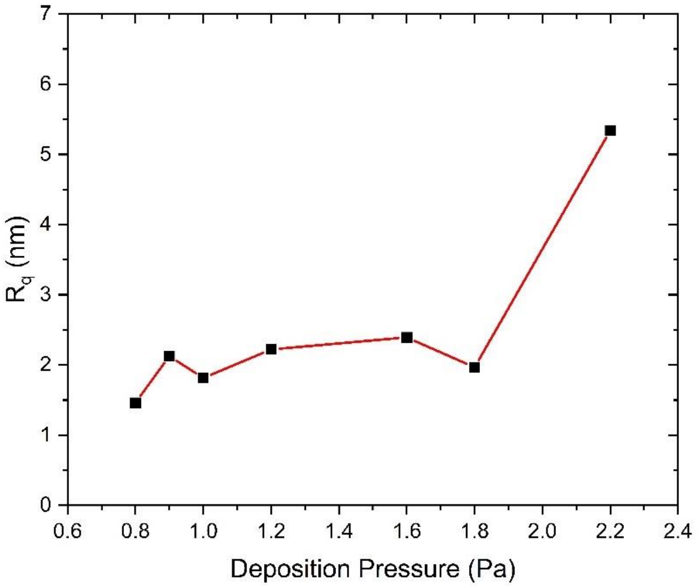
Experimental plan

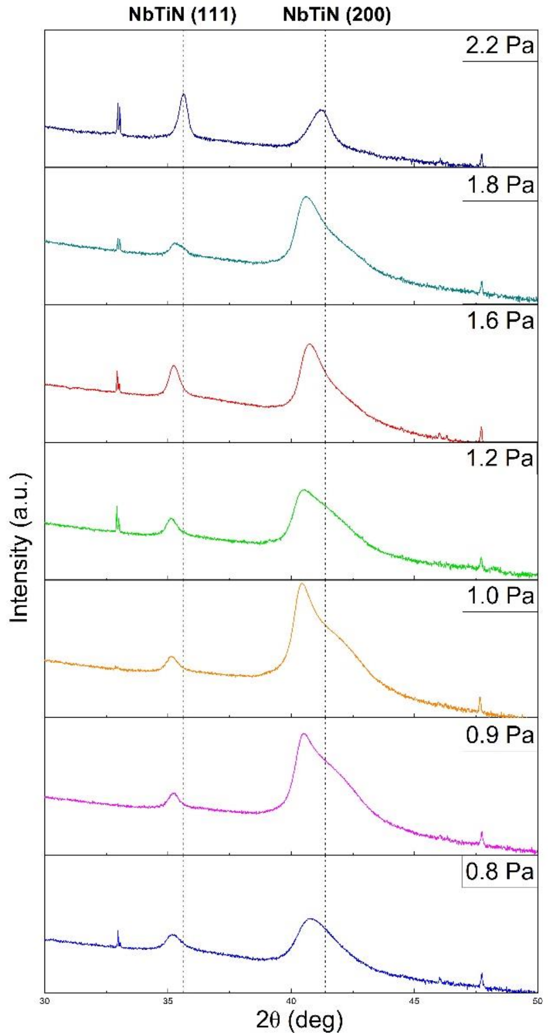


Effect of deposition pressure

Deposition conditions:
P_{target} = 400 W, N_2 = 9%,
Bias = -50V, d.c. = 10%



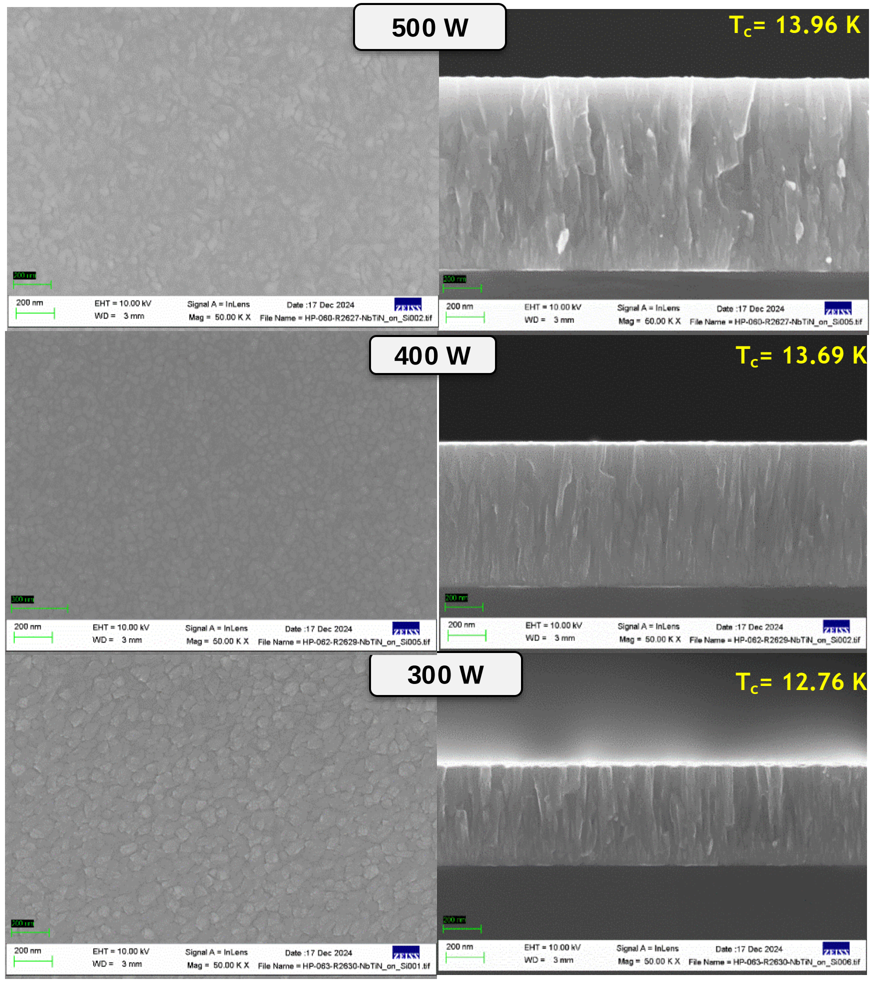


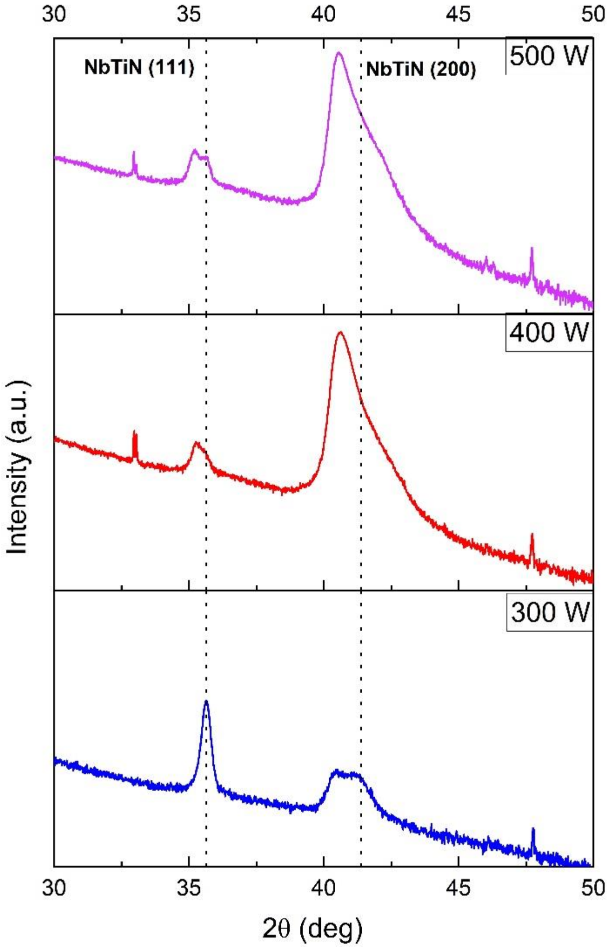


HiPIMS-NbTiN

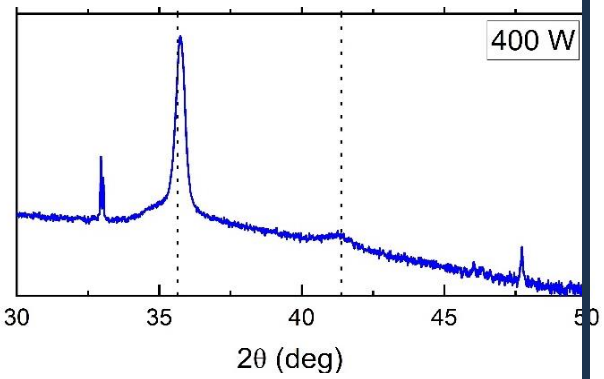
Effect of cathode power

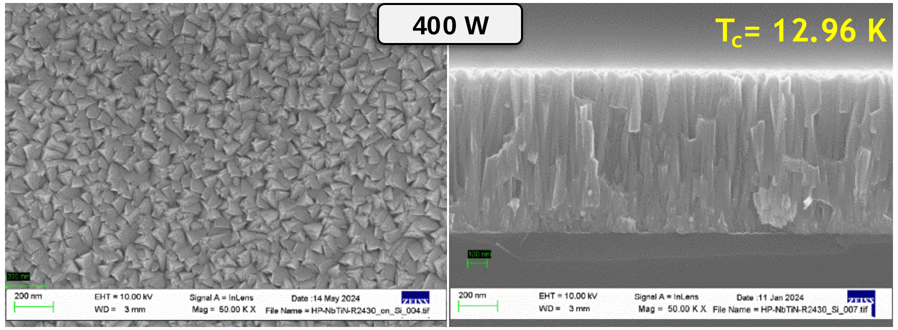
Deposition conditions:
N_2 = 9%, Bias = -50V, p_{dep} = 1.8 Pa, d.c. = 10%





d.c. = 20%

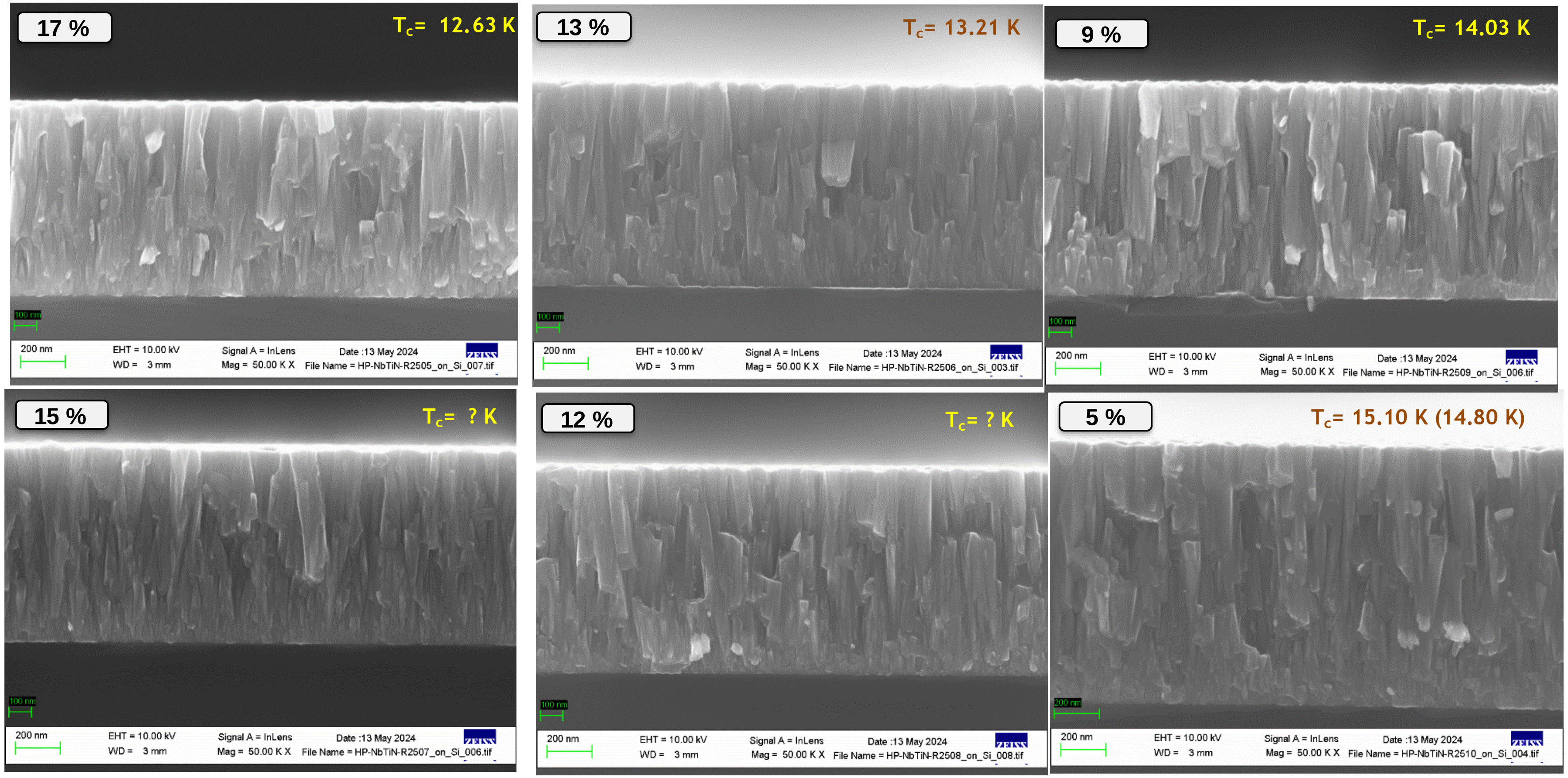


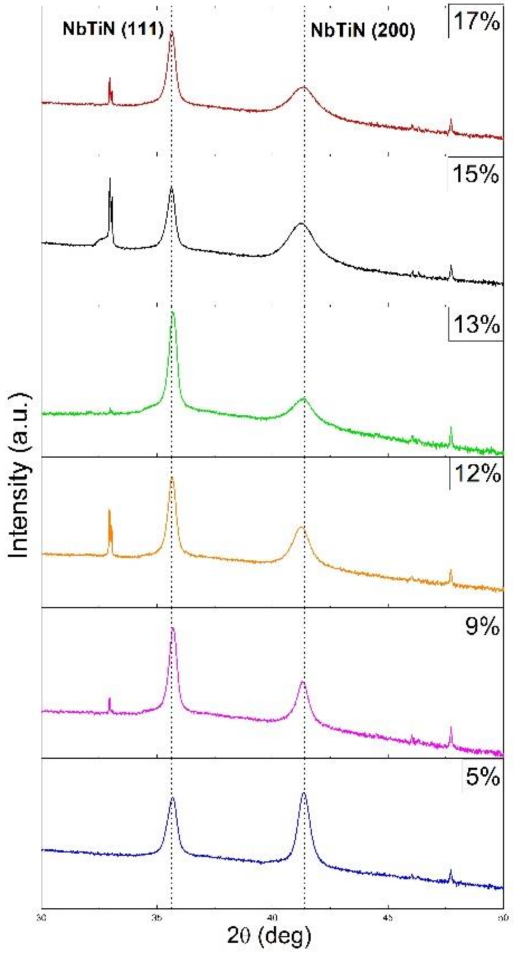


HiPIMS-NbTiN

Effect of N_2 concentration

Deposition conditions:
P_{target} = 400 W, p_{dep} = 1.6 Pa, Bias = -50V, **d.c. = 20%**

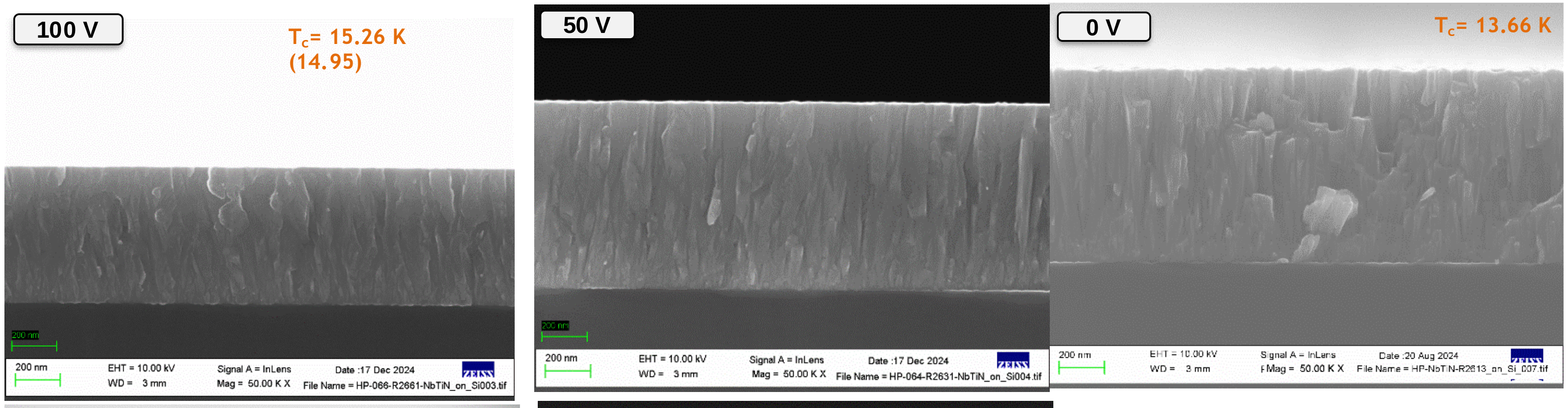


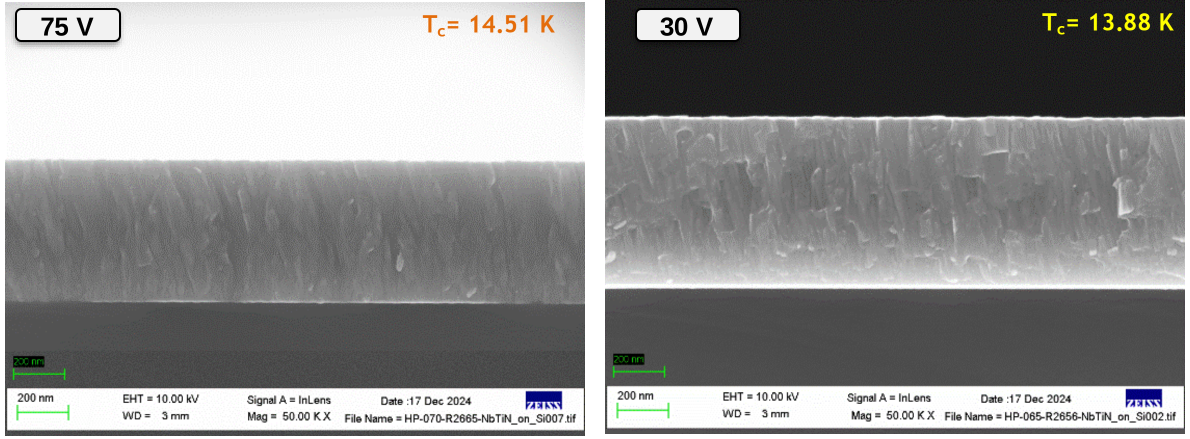


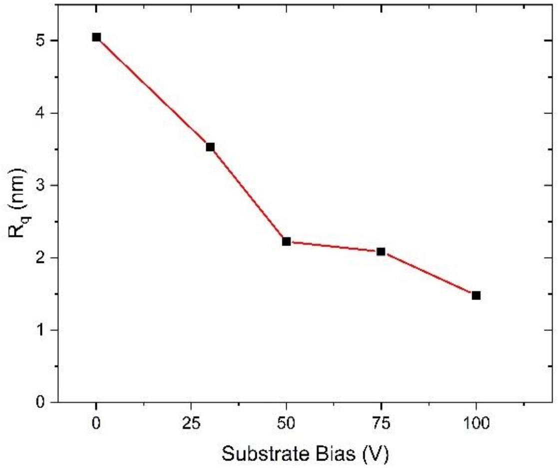
HiPIMS-NbTiN

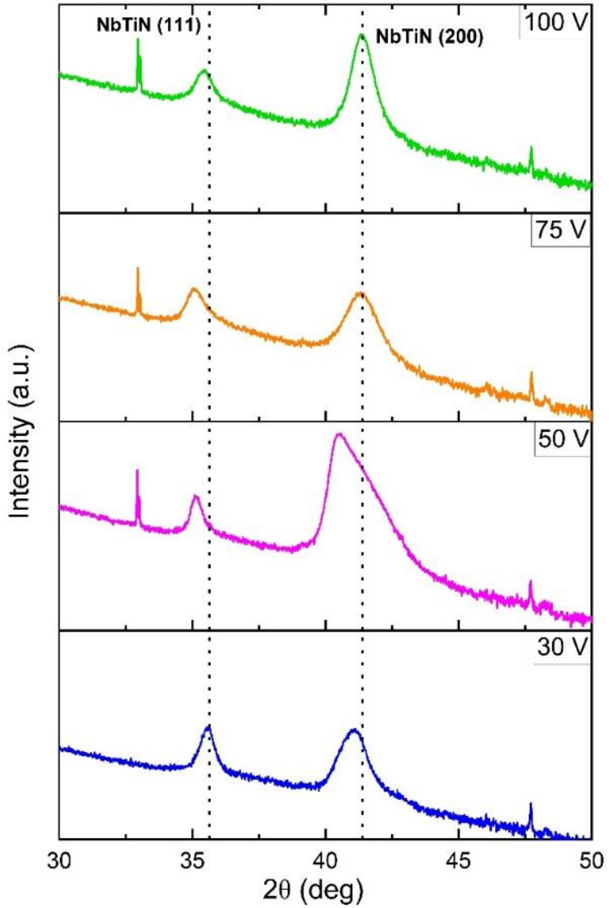
Effect of substate bias

Deposition conditions:
P_{target} = 400 W, p_{dep} = 1.2 Pa, N_2 = 9%, **d.c. = 10%**









HiPIMS-NbTiN

Cathode Power Study

- 400 W Cathode power provides excellent process stability without stress-related film damage, even at lower deposition pressures (~ 0.8 Pa)
- High cathode powers (500 W, 600 W…) exhibit high Tc, but stress-related film damage occurs, especially at lower deposition pressures (~ 1.2 Pa)
- **More studies ?** - Measuring T_c values for the films deposited at higher cathode powers (with higher deposition pressures (~1.8 Pa)) – May not provide higher T_c films
- **Conclusion**: 400 W cathode might be an ideal choice (especially for the lower deposition pressures that typically give higher Tc values)

Deposition Pressure Study

- Lower deposition pressures generally lead to higher Tc . However, it appears that reducing the pressure below 1.6 Pa does not significantly impact the Tc values.
- **More Stuides:** To confirm this observation, Tc measurements should be conducted for films deposited at lower pressures (1.0 Pa, 0.9 Pa, and 0.8 Pa).
- **Conclusion:** Lower deposition pressures might be an ideal choice; however, further Tc measurements at deposition pressures below 1.6 Pa are required for confirmation.

Duty Cycle Study

- Lower duty cycles result in higher Tc , as observed in coatings deposited at 1.6 Pa and 1.8 Pa. However, at 1.3 Pa, the deposition pressure does not appear to influence Tc values.
- **More Studies:** To confirm this trend, Tc measurements are required for the remaining films with duty cycles of 12%, 15%, and 17%.
- **Conclusion:** A lower duty cycle appears to be an ideal choice, but further analysis is necessary for validation, especially at lower deposition pressures.

Substrate Bias Study

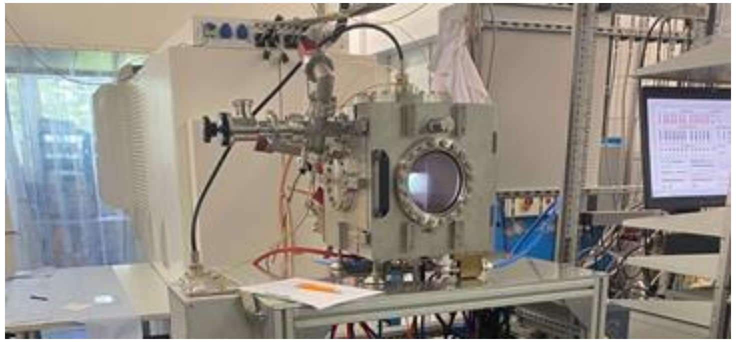
- The substrate bias has a significant influence on Tc, with Tc increasing as the substrate bias increases.
- **More Studies:** Tc measurements at substrate biases of 125 V and 150 V would provide further insights into this trend.
- **Conclusion:** Higher substrate bias appears to be beneficial for achieving higher Tc values.

Nitrogen Flow Study

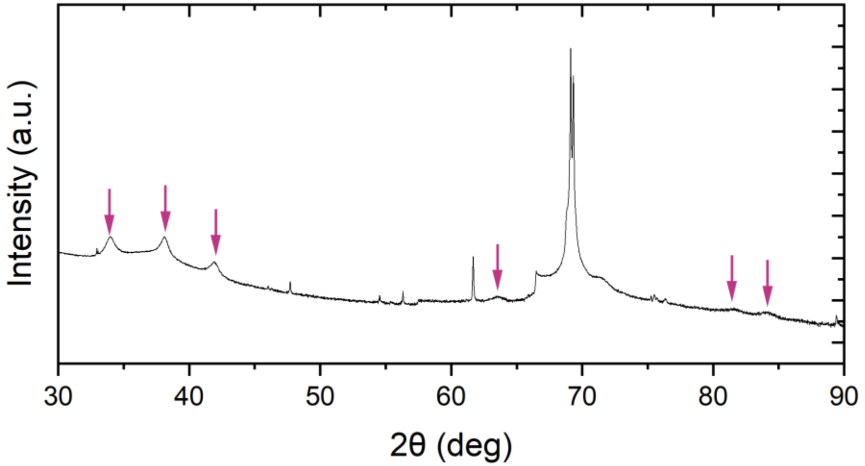
- The N2 flow has a significant influence on Tc , with Tc increasing as the N2 flow decreases. Currently, Tc measurements are available only for films deposited with a 20% duty cycle.
- **More Studies:** Tc measurements for films deposited with a 10% duty cycle are required.
- **Conclusion:** Lower N2 flow appears to be beneficial for achieving higher Tc values.

Nb_3Sn RF sputtering

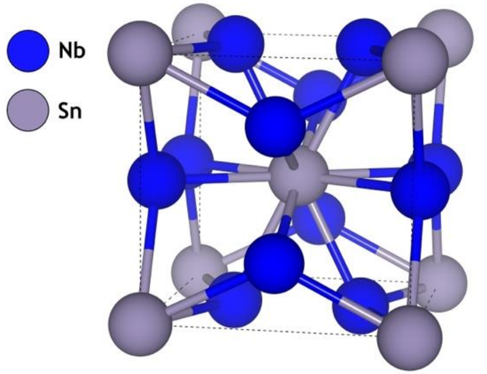
Nb_3Sn in BoxCoater



NbSn_03: p = 1 Pa, P =100W, T = 580°C



***P* m-3n**



Test depositions for multilayer systems

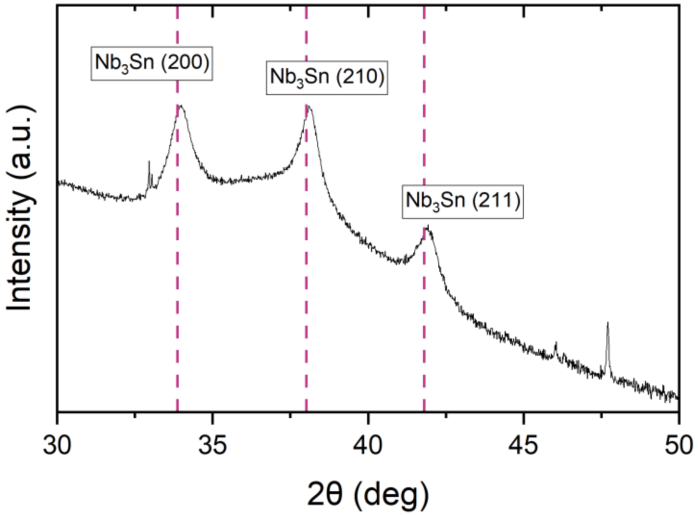
Later process transfer to CC800 – HiPIMS

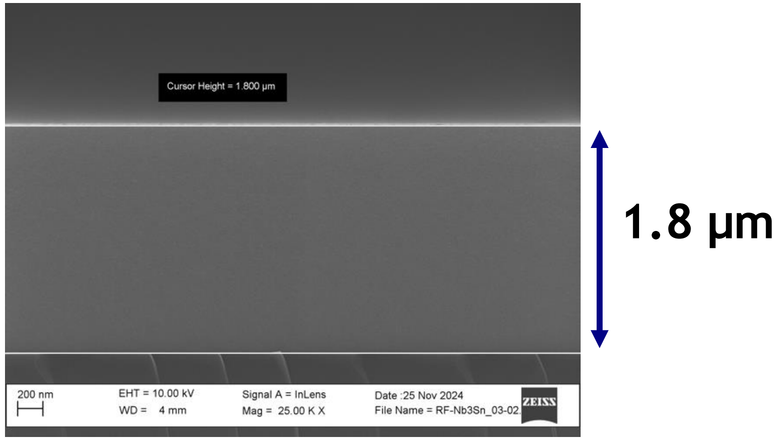
Parameters:

T_{dep} → 550°C - 650°C

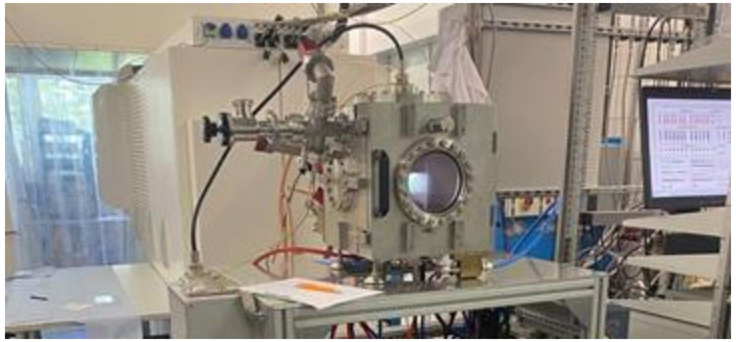
P(cathode) → low, <1 W/cm²

p_{dep} = 1 Pa (Ar) → must be Kr



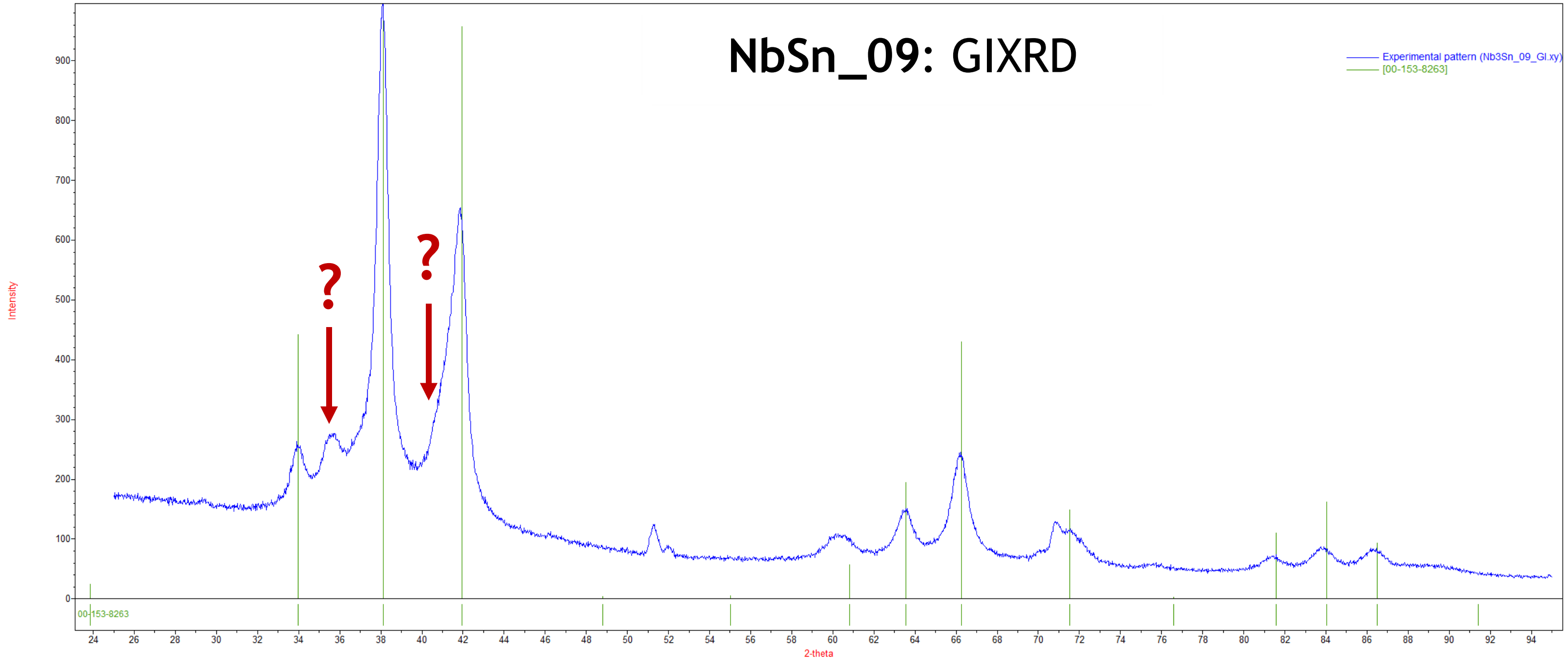


Nb_3Sn in BoxCoater



Test depositions for multilayer systems
Later process transfer to CC800 – HiPIMS

Parameters:
T_{dep} → 550°C - 650°C
P(cathode) → low, <1 W/cm² (<100W)
p_{dep} = 1 Pa (Ar) → must be **Kr**

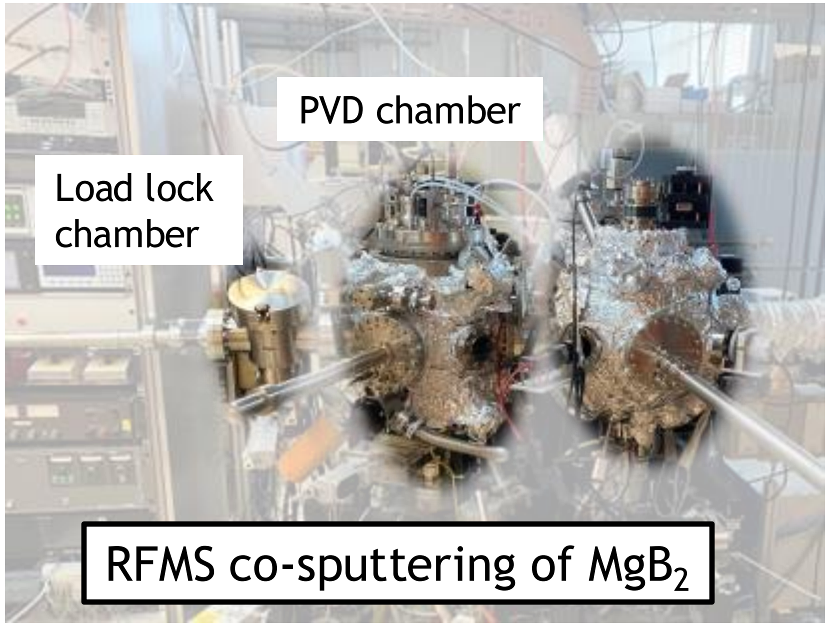


NbSn_07: p = 0.6 Pa, P =100W → a = 5.288(1) Å
NbSn_09: p = 1 Pa, P = 150W → a = 5.285(2) Å

EDX: 86% Nb and 14% Sn !
Not superconducting!

co-MgB_2: RF sputtering

MgB_2 in PVD/SEY chamber

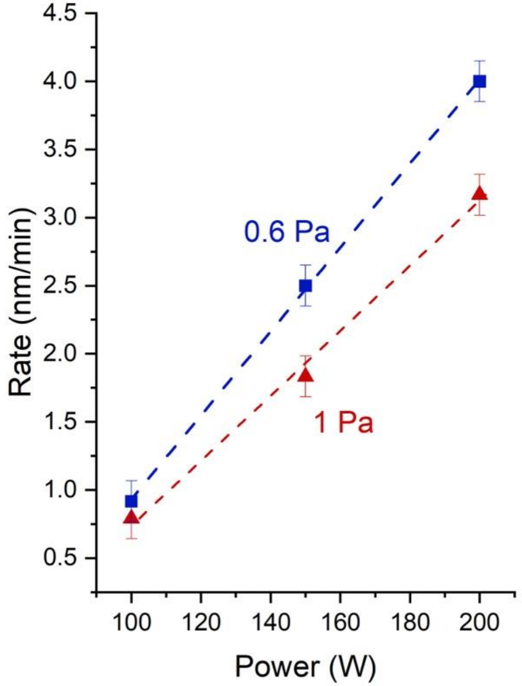


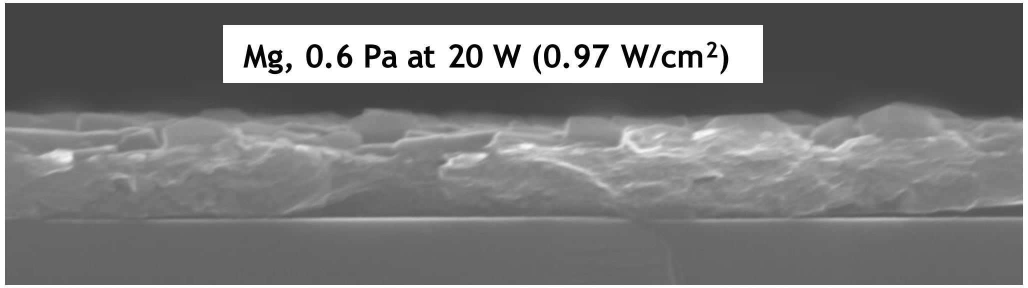
Adjustment of Mg and B deposition rates *R* (nm/min):

Variation of the cathode power and deposition pressure

$R_{Mg} : R_B = 3 : 2$ to get MgB_2

- Substrate temperature: 300°C – 600°C ?
- Ar pressure: 0.5 – 1 Pa
- Deposition at RT and annealing?





Plans and outlook

- Limit for **DC-NbTiN** ? Need higher substrate temperature?
- Optimization of **HiPIMS-NbTiN** deposition: low p_{dep}, low d.c., substrate bias…?
- Deposition of multilayer (SS or SIS) structures with
  DC-NbTiN (“best” conditions) / DC-AlN/ **HiPIMS-Nb** or **bulk Nb**
- Start of **MgB_2** deposition in PVD/SEY deposition chamber by RF co-sputtering: **promising?**
- Deposition of **Nb_3Sn** in BoxCoater with **Kr**: test for the multilayer structures
- QPR samples for RF test and surface resistance

THANK YOU FOR YOUR ATTENTION!